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**EUROPEAN PATENT APPLICATION**

② Application number: 88310943.1

⑤ Int. Cl.<sup>4</sup>: G 01 N 11/16

②② Date of filing: 18.11.88

③① Priority: 18.11.87 JP 289521/87

④3 Date of publication of application:  
24.05.89 Bulletin 89/21

⑧ Designated Contracting States:  
CH DE FR GB IT LI SE

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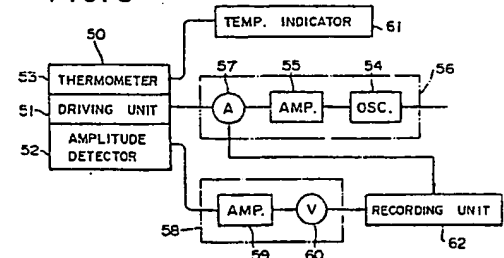
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(54) **Vibration-type rheometer apparatus.**

57) A vibration-type rheometer including a vibration-type viscometer having a pair of vibrator subassemblies (4) which resonate as in a tuning fork. The pair of vibrator subassemblies (4) constituting a tuning fork vibrator each has at its free end a sensor plate (9) formed from a thin metal plate placed into a sample (35) to be measured and are driven at the same frequency with phases opposite by electromagnetic driving means (12, 13) together with the sensor plate (9). A control unit (56) is provided for supplying a driving current having a magnitude which changes steplessly and continuously to the electromagnetic driving means (12, 13) in order to change with time the vibration-inducing force in the pair of vibrator subassemblies (4). The amplitude of vibration of the vibrator subassemblies (4) which changes with the viscous resistance received by the sensor plates from the sample is electrically detected, and the detected value is sent to the recording means (62) together with the value of the driving current to thereby indicate the behaviour of the sample (35) with respect to the external force which changes by time.

FIG. 3



## Description

## VIBRATION-TYPE RHEOMETER APPARATUS

The present invention relates to a rheometer for measuring the phenomenon of the deformation and flow of a fluid, and more particularly, to a vibration-type rheometer including a pair of tuning fork-like members capable of being vibrated in a sample.

The flowability of simple liquids, for example, such as water, alcohol, glycerine or the like differs with viscosity, but these liquids exhibit a Newtonian viscosity, that is, a straining rate proportional to a stress during the flow. On the other hand, it is known that thick liquids having a relatively complicated construction, for example such a paint, toothpaste, mayonnaise and cold cream may exhibit a non-Newtonian viscosity in which the flow will not start unless an external force exceeds a predetermined value. Another phenomenon is a property of gels which change into sol when subjected to shaking or vibration, and then return again to gels when left standing which is called thixotropy.

A measurement of Newtonian viscosity or non-Newtonian viscosity, and particularly the degree of thixotropy can be found by evaluating the area of a hysteresis loop which appears with the revolution of a rotary viscometer. In the rotary viscometer, the viscosity is obtained by rotating a cylindrical body in viscous fluid and measuring the torque due to the viscosity exerted on the cylindrical body. In a coaxial double cylindrical meter, a fluid is put between an inner tube and an outer tube, and the torque exerted on the inner tube when the outer tube is rotated is measured. This measurement is carried out by hanging the inner tube from a torsion wire and measuring a torsional angle of the torsion wire. If the design is such that the angular velocity of the rotary body is made variable so as to vary a shear rate corresponding thereto the viscometer can be applied to the measurement of the flow characteristics of non-Newtonian viscous fluids.

However, it is necessary to vary the shape of a rotary body depending on the sample to be measured. Furthermore, since it is cumbersome to clean and wash the rotary body after use, this gives rise to a problem in handling. In addition, since the viscometer is affected by the inertia of the rotary body or by the flow of the sample, the range of controllable angular velocity of the rotary body is narrow, and various measuring patterns cannot be selected.

The present invention is concerned with a vibration-type rheometer apparatus including a viscometer having a pair of tuning-fork members capable of being vibrated in a sample. A vibration-type viscometer having a pair of tuning fork-like members is known from U.S. Patent No. 4,602,505 (corresponding to European Patent No. 112,156) entitled "APPARATUS FOR MEASURING VISCOSITY" issued to the present inventors on July 29, 1986. Also, an improved vibration-type viscometer of this type has been proposed in U.S. Patent No. 4,729,237 (corresponding to European Patent 233,408) entitled "TUNING FORK VIBRATION-TYPE VISCOSITY

MEASURING APPARATUS" issued to the present inventors on March 8, 1988. These vibration type viscometers each comprise vibrator means of a tuning fork including a pair of vibrator subassemblies, each vibrator subassembly having at its free end a sensor plate to be inserted into a sample to be measured, a driving unit for applying vibrations to said pair of vibrator subassemblies, and a detector for detecting the vibration amplitude of said pair of vibrator subassemblies which changes due to a viscosity resistance applied to the sensor plates when placed in the sample and for converting the vibration amplitude into an electric signal. The driving unit comprises a combination of an electromagnetic coil and a permanent magnet, in which the pair of vibrator subassemblies are vibrated in a phase opposite to each other, that is, a phase difference of 180 degrees under the same frequency. In the vibration-type viscometers so far proposed, the driving frequency is 30 Hz, with a one-side amplitude with no-load of 20 microns, which is constant.

According to the present invention there is provided a vibration-type rheometer apparatus for measuring the behaviour of a fluid sample with respect to an external force with changes with time, the apparatus comprising:

a. a support block firmly secured to a base frame, said support block having a support column at the lower part thereof;

b. tuning fork vibrator means including a pair of vibrator subassemblies secured to said support block, extending downwardly from said support block and being arranged on opposite sides of said support column, said vibrator subassemblies each having a thin and flat sensor plate to be placed into said sample to be measured at a free end thereof, both said sensor plates being disposed in the same imaginary vertical plane;

c. means for driving said pair of vibrator subassemblies with phases opposite to each other at the same driving frequency;

d. means for detecting the amplitudes of vibration of said pair of vibrator subassemblies which change with the viscous resistance received by said sensor plates placed in said sample, said means outputting an electric signal representative of said amplitude;

e. control means for supplying a driving current having a magnitude which steplessly and continuously changes to said driving means to change with time the vibration-inducing force in said pair of vibrator subassemblies and

f. recording means for plotting, with respect to the change in magnitude of said driving current, an output value of said detection means which changes in response thereto.

Since a continuous change in the magnitude of the driving current applied to the driving unit results in a continuous change in the vibration inducing force to the pair of vibrator subassemblies, if the

change in the amplitude in response to the change in the vibration-inducing force to the pair of vibrator subassemblies is continuously detected then it is possible to measure an amount representative of the rate of change with time in the motion of a fluid. In this case, in contrast with the rheometer using a conventional rotary viscometer for generating a concentric circular flow in the fluid, merely slight vibrations are generated by the pair of vibrator subassemblies according to the present invention, and therefore the measuring pattern resulting from the control of the way of applying the vibration-inducing force and the magnitude may have considerable freedom. The control of the driving current in typical examples of the measuring pattern are as follows :

Pattern 1: From time  $t_0$  to  $t_1$ , steplessly, and continuously increased, and then from  $t_1$  to  $t_2$ , steplessly and continuously decreased.

Pattern 2: Similarly to the pattern 1, continuously increased until the time  $t_1$ , and held constant after the time  $t_1$ .

Pattern 3: Similarly to the pattern 1, continuously increased until the time  $t_1$ , and thereafter held constant until the next time  $t_2$ , and then cut off to 0.

Pattern 4: Similarly to the pattern 1, continuously increased until the time  $t_1$ , and conversely steplessly and continuously lowered from the time  $t_1$  to the time  $t_2$ , these ups and downs being repeated.

These measuring patterns can be obtained easily by program-controlling the driving current.

According to the present invention, the driving current to the driving unit of the pair of vibrator subassemblies is steplessly and continuously changed and the change in the amplitude value response to the change in the vibrator-subassemblies is continuously detected whereby the rheology of the fluid can be measured. Therefore, the measurement can be simply conducted without giving rise to the problems in handling encountered in the use of a conventional rotary viscometer.

An embodiment of the invention will now be described, by way of example only, with reference to the accompanying drawings in which :

FIG. 1 is a partial cut-away side elevational view of a vibration-type viscometer used in the rheometer of the present invention;

FIG. 2 is a side view showing essential parts shown in FIG. 1 in an exploded form;

FIG. 3 is a block diagram illustrating an amplitude rheometer apparatus according to the present invention;

FIGS. 4, 5 and 6 are respectively explanatory view showing the flow characteristics of a sample measured by the vibration-type rheometer apparatus according to the present invention, FIG. 4 is of the case where a sample is mayonnaise, FIG. 5 is of the case where a sample is cold cream, and FIG. 6 is of the case where a sample is milky liquid ; and

FIG. 7 is an explanatory view representative of various measuring patterns.

First, referring to FIG. 3, a vibration-type rhe-

ometer apparatus according to the present invention includes a vibration-type viscometer generally indicated at 50. This vibration-type viscometer comprises an electromagnetic driving unit 51, an amplitude detector 52 and a thermometer 53, which will be described in detail later. The electromagnetic driving unit 51 has a permanent magnet 13 (FIG. 1) and an electromagnetic coil 12 (FIG. 1) cooperated therewith, the electromagnetic coil 12 receiving a driving current having a magnitude which steplessly and continuously changes from a control unit 56 including an oscillator 54 and a variable amplifier 55. This control unit 56 is provided with an ammeter 57 for measuring the magnitude of a driving current supplied to the electromagnetic driving unit 51. The amplitude detector 52 comprises, for example, a non-contact system eddy current loss detection type displacement detector 14 (FIG. 1) and an output signal of this detector 14 is sent to an amplitude display unit 58. This amplitude display unit 58 includes an amplifier 59 yielding a voltage value. A thermometer 53 has a temperature probe 21 (FIG. 1), an output signal of which is supplied to a temperature indicator 61. A value measured by the ammeter 57 and a value measured by the voltmeter 60 for measuring the output of the amplifier 59 yielding a voltage value. A thermometer 53 has a temperature probe 21 (FIG. 1), an output signal of which is supplied to a temperature indicator 61. A value measured by the ammeter 57 and a value measured by the voltmeter 60 are sent to a recording unit 62 such as an X-Y recorder, and the recording unit 62 plots the detected voltage which is representative of a change in the amplitude value from the amplitude detector 52 corresponding to the driving current sent to the electromagnetic driving unit 51 the magnitude of which continuously and steplessly changes.

The vibration-type viscometer per se used in the present invention is of the general type disclosed in U.S. Patent Nos. 4,602,505 and 4,729,237. Referring to FIG. 1, the vibration-type viscometer is provided with a hollow support block formed of a rigid material firmly secured to a frame shaft 1 extending from a base (not shown), the support block 2 having a downwardly extending support column 3. A pair of vibrator subassemblies 4 constituting tuning fork-like vibrations are secured to the lower end of the support block 2, and these vibrator subassemblies 4 downwardly extend from the support block 2 and occupy opposite sides of the support column 3. The vibrator subassemblies 4 each include a leaf spring 5 with one end secured to the support block 2 by means of a screw 6 through a stop 7, a long intermediate plate 8 firmly mounted on the other end of the leaf spring 5, and a sensor plate 9 secured to the end of the intermediate plate 8 by means of a screw 10. The leaf spring 5 is preferably made of constant elastic spring steel, and the intermediate plate 8 is preferably made of a light relatively rigid material, for example, such as aluminium. The sensor plate 9 is preferably made of stainless steel which is as thin as 0.2 mm or so, is flat and has a chemical resistance, the sensor plate having a free end formed into a disk 11 having a diameter of 20

mm or so, for example.

One vibrator subassembly 4 is arranged symmetrically with the other vibrator assembly, and a permanent magnet 13 cooperated with a pair of electromagnetic coils 12 mounted on the support column 3 is provided on the intermediate plate 8. The combination of the electromagnetic coils 12 and the permanent magnet 13 functions as a driving device 51 for vibrating the corresponding vibrator subassemblies 4. The driving device 51 being supplied with a driving current having a magnitude which steplessly and continuously changes from the control unit 56 (FIG. 3) as described above to thereby vibrate the pair of vibrator assemblies 4 in phases opposite to each other, that is, a phase difference of 180 degrees under the same frequency in response to a vibration-inducing force having a magnitude which steplessly and continuously changes. According to a preferred example, the driving frequency is 30 Hz, and the driving current linearly changes from 0 to 1000mA. A pair of sensor plates 9 are disposed within the same imaginary vertical plane, and as a result, a torsional reaction in the support block 2 generated in the case where the sensor plates are disposed in different imaginary vertical planes can be avoided. While the relative arrangement of the electromagnetic coils 12 and the permanent magnet 13 may be reversed, the provision of the electromagnetic coils 12 on the side of the support column 3 as in the example shown in the drawing is suitable in that a lead wire 15 of the coil 12 can be guided to a metal terminal 16 (upward) passing through the support column 3.

The support column 3 between the support block 2 and the electromagnetic coil 12 is provided with a displacement detector 14 so as to oppose to a leaf spring 5 of the vibrator subassembly 4, the displacement detector 14 converts the amplitude of one vibrator subassembly 4 into an electric signal. In this case, a further displacement detector may be provided for the other vibrator subassembly, but since both the subassemblies 4 exhibit substantially the same amplitude, then one will therefor suffice. When a pair of sample plates 9 are placed into a sample as will be described later, the amplitude of the vibrator subassemblies 14 is affected by the change in the viscous resistance, and therefore the displacement detector 14 electrically detects the amplitude, and the viscosity of the sample is arithmetically calculated from the detected value in a well known manner. The displacement detector 14 can be, for example, of a well known non-contact system eddy current loss detector but in the case where this well known displacement is used, the leaf spring 5 opposed thereto is formed of a magnetic steel spring. A well known optical displacement sensor can also be used in place of an eddy current loss detection type displacement sensor. A lead wire 17 of the displacement detector 14 is also guided to a common terminal element 16 passing through the support column 3.

Turning to FIG. 2, a thermometer generally indicated at 20 is mounted on the lower end of the support column 3, and a sheathed probe 21 of the thermometer 20 extends downward. This tempera-

ture probe 21 occupies a position between the pair of sensor plates 9 and is disposed in the same imaginary vertical plane, the probe 21 having its lower end disposed in substantially the same imaginary horizontal plane as the pair of the sensor plates 9. Since the temperature probe 21 is aligned in the same imaginary vertical plane as the pair of sensor plates 9, the occurrence of sample turbulence due to the presence of the temperature probe 21 between the sensor plates is prevented. The thermometer 20 can be a well known type, for example, in which a platinum temperature measuring resistance is provided within a sheath, which well known thermometer has a circuit unit 22 including an amplifier at the base end of the sheath. A lead wire 23 of the circuit unit 22 reaches a common terminal element 16 passing through the support column 3.

An external thread 30 is formed at the lower end of the support column 3, and a carrier device 32 having an adjusting nut member 31 threadedly engaged with the external thread 30 mounted on the support column 3. The carrier device 32 detachably carries a sample container 33, and functions as a lid for closing an open surface of the sample container 33. The sample container 33 is conveniently made from a transparent glass like a beaker. This container has a flange 34 around the open edge thereof, and has an index comprising two parallel lines representative of an allowable amount of a sample 35 to be placed therein. The carrier device 32 includes a lid member 37 formed of a synthetic resin which is excellent, for example, in its heat insulating properties and having a size just fitted into the sample container 33, said lid member 37 having a flange 38. The lid member 37 is provided with a pair of well known clamp elements 39, and when these clamp elements 39 are brought into engagement with the flange 34 of the sample container 33, the sample container 33 can be mounted on the carrier device 32. The adjusting nut member 31 threadedly engaged with the external thread 30 of the support column 3 has a stopper 40 at the lower part thereof, and the axial movement is restricted by the stopper 40. The lid member 37 is formed with a hole 41 through which the lower end of the support column 3 may pass and a pair of head-diffusion preventive narrow slits 42 through which a pair of sensor plates 9 may pass.

Normally, the carrier device 32 is mounted on the lower end of the support column 3, and the sample container 33 is detachably mounted on the carrier device 32. Two pins 44 are downwardly secured to the lower end of the support column 3, the pins 44 being located on the sides opposite to each other of the temperature probe 21, occupying a position between the probe and the sensor plate 9 and being aligned in the imaginary vertical plane in which sensor plates 9 and the temperature probe 2 are distributed. The pin 44 has its extreme end which has a function as an indicator representative of a desirable surface level of the sample 35 within the container 33. More specifically, if a misalignment is present between the extreme end of the pin 44 and the surface level of the sample 35, the adjusting nut member 31 of the carrier device 32 is rotated to axially move the sample container 33 along with the

carrier device 32 toward the support column 3, whereby the misalignment as described above can be overcome. As a result, even if the sample differs in amount within the allowable range between the two index lines 36 provided on the sample container 33, the sensor plate 9 and the temperature probe 21 can be always inserted by a predetermined length into the sample without involving the trouble of ensuring that the sample has a strictly determined amount and errors in measurement caused by a difference in the depth to which the sensor are inserted are avoided. FIGS. 4, 5 and 5 are respectively graphs showing the results obtained by measuring the flow characteristics of three kinds of samples using a vibration-type rheometer manufactured in accordance with the preferred example of the present invention. In these graphs, the axis of ordinate indicates the magnitude I of a driving current corresponding to a vibration-inducing force applied to the pair of vibrator subassemblies 4 while the axis of the abscissa indicates the magnitude E of the detected voltage corresponding to the amplitude of vibration of the pair of vibrator subassemblies 4. FIG. 4 is of the case where the sample is mayonnaise, FIG. 5 is of the case where the sample is cold cream, and FIG. 6 is of the case where the sample is milky liquid. According to FIGS. 4 and 5, the curve obtained by gradually increasing the vibration-inducing force and the curve obtained by gradually decreasing it conversely depict a hysteresis loop, from which can be learned that these samples exhibit a thixotropic non-Newtonian viscosity. According to FIG. 6, it has been found that the change in the amplitude with respect to the change in the vibration-inducing force is linear, and this sample is a material exhibiting a Newtonian viscosity.

FIG. 7 is a graph explaining the variation of driving current in various measuring patterns, in which graph, the axis of ordinate indicates the current value I and the axis of abscissa indicate the time T. The status of the measuring patterns represented by these graphs are as follows:

FIG. 7a - pattern 1: From time  $t_0$  to  $t_1$ , increased steplessly and continuously, and then from  $t_1$  to  $t_2$ , decreased steplessly and continuously.

FIG. 7b - pattern 2: Similarly to the pattern 1, increased continuously until time  $t_1$ , and held constant after the time  $t_1$ .

FIG. 7c - pattern 3: Similarly, increased continuously until time  $t_1$ , thereafter held constant till time  $t_2$ , and then cut off to 0.

FIG. 7d - pattern 4: Similarly, increased continuously until time  $t_1$ , conversely lowered steplessly and continuously from time  $t_1$  to time  $t_2$ , these up and down being repeated.

The aforementioned measuring patterns merely comprise specific embodiments, and according to the present invention, various modifications can be made without limit to these measuring patterns.

In at least the preferred embodiment the present invention provides a new rheometer apparatus which eliminates the disadvantages noted above with respect to a rheometer using a conventional

rotary viscometer.

In at least the preferred embodiment the present invention also provides a rheometer which can widely change the way of applying an external force to a sample to be measured and the strain rate so that various measuring patterns may be selected.

In at least the preferred embodiment the present invention also provides a rheometer which is simple in handling and easy in operation.

## Claims

1. A vibration-type rheometer apparatus for measuring the behaviour of a fluid sample (35) with respect to an external force which changes with time, the apparatus comprising :

a. a support block (2) firmly secured to a base frame (1), said support block (2) having a support column (3) at the lower part thereof;

b. tuning fork vibrator means including a pair of vibrator subassemblies (4) secured to said support block (2), extending downwardly from said support block (2) and being arranged on opposite sides of said support column (3), said vibrator subassemblies (4) each having a thin and flat sensor plate (9) to be placed into said sample (35) to be measured at a free end thereof, both said sensor plates (9) being disposed in the same imaginary vertical plane;

c. means (12, 13) for driving said pair of vibrator sub-assemblies (4) with phases opposite to each other at the same driving frequency;

d. means (14) for detecting the amplitudes of vibration of said pair of vibrator subassemblies (4) which change with the viscous resistance received by said sensor plates (9) placed in said sample (35), said means outputting an electric signal representative of said amplitude;

e. control means (56) for supplying a driving current having a magnitude which steplessly and continuously changes to said driving means (12, 13) so as to change with time the vibration-inducing force in said pair of vibrator subassemblies; (4) and

f. recording means (62) for plotting, with respect to the change in magnitude of said driving current, an output value of said detection means which changes in response thereto.

2. The vibration-type rheometer apparatus according to claim 1, wherein said control means (5) includes an oscillator (54) having a fixed frequency, a variable amplifier (55) for forming a driving current having a magnitude which steplessly and continuously changes from an output of said oscillator, and an ammeter (57) for measuring the magnitude of

said driving current.

3. The vibration-type rheometer apparatus according to claims 1 or 2, wherein said control means (56) program-controls the magnitude of said driving current.

4. The vibration-type rheometer apparatus according to claim 3, wherein said control means (56) controls the magnitude of said driving current in a manner such that the magnitude is steplessly and continuously increased from time  $t_0$  to  $t_1$ , and held constant after the time  $t_1$ .

5. The vibration-type rheometer apparatus according to claim 3, wherein said control

means (50) controls the magnitude of said driving current in a manner such that the magnitude is steplessly and continuously increased from time  $t_0$  to  $t_1$ , and held constant from the time  $t_1$  and  $t_2$ , and then cut off to 0.

6 The vibration-type rheometer apparatus according to claim 3, wherein said control means (56) controls the magnitude of said driving current in a manner such that the magnitude is steplessly and continuously increased from time  $t_0$  to  $t_1$ , and steplessly and continuously lowered from the time  $t_1$  to time  $t_2$ .

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FIG. 1

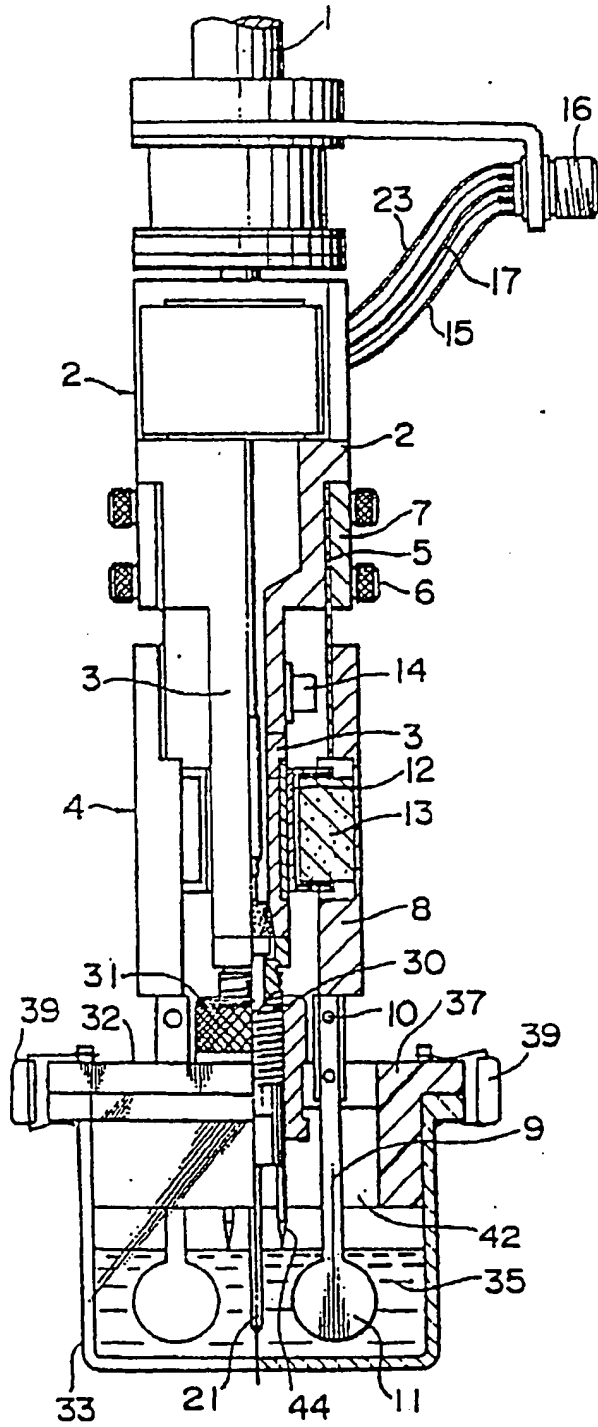


FIG. 2

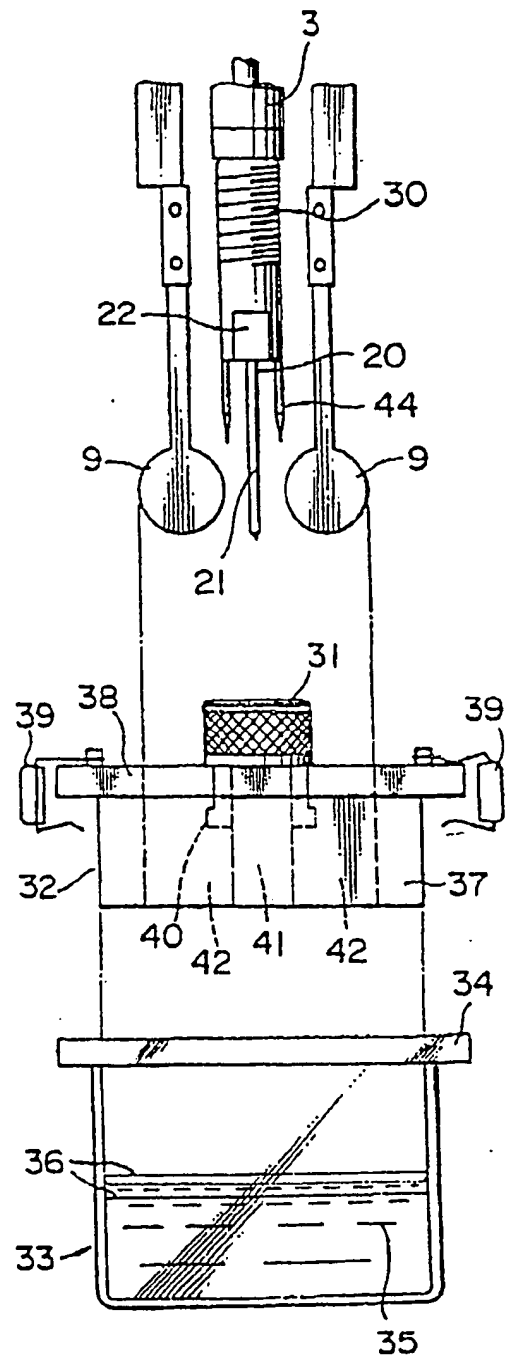


FIG. 3

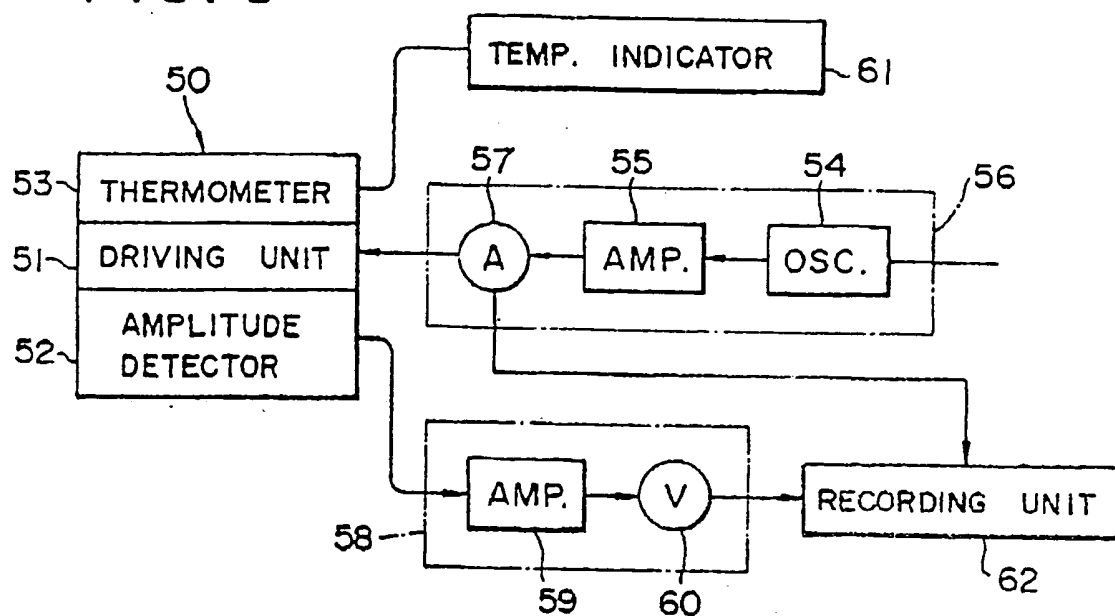


FIG. 7a

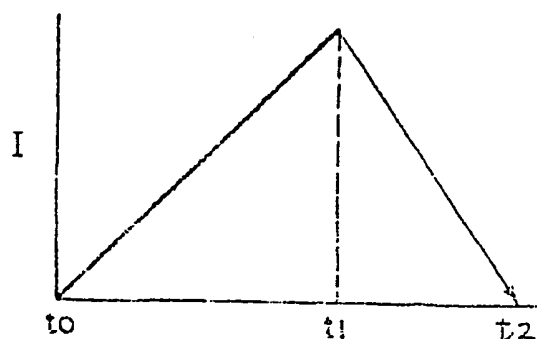


FIG. 7b

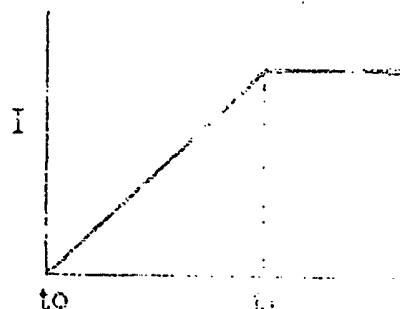


FIG. 7c

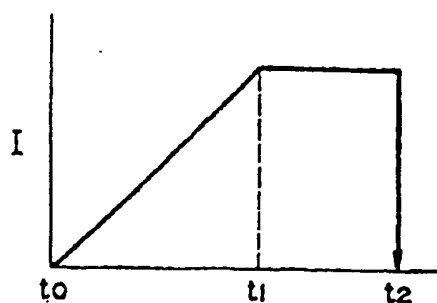


FIG. 7d

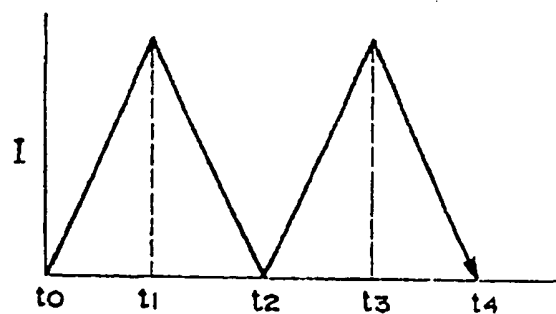


FIG. 4

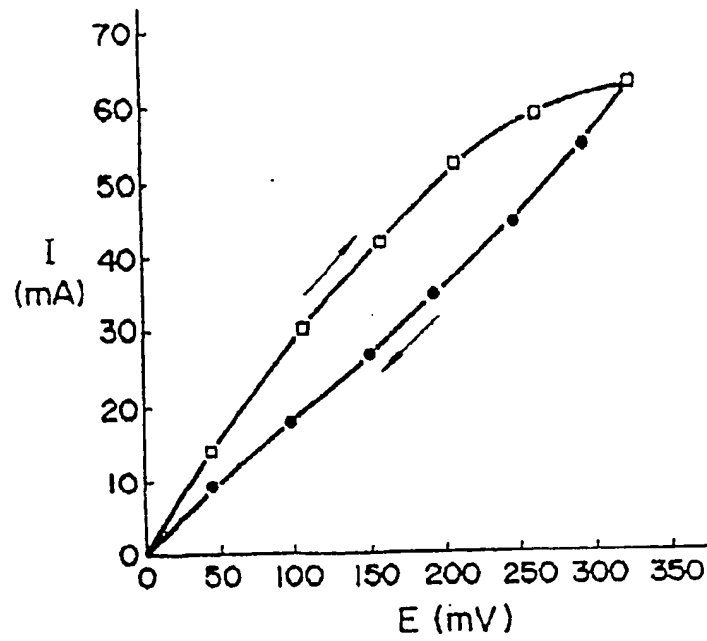


FIG. 5

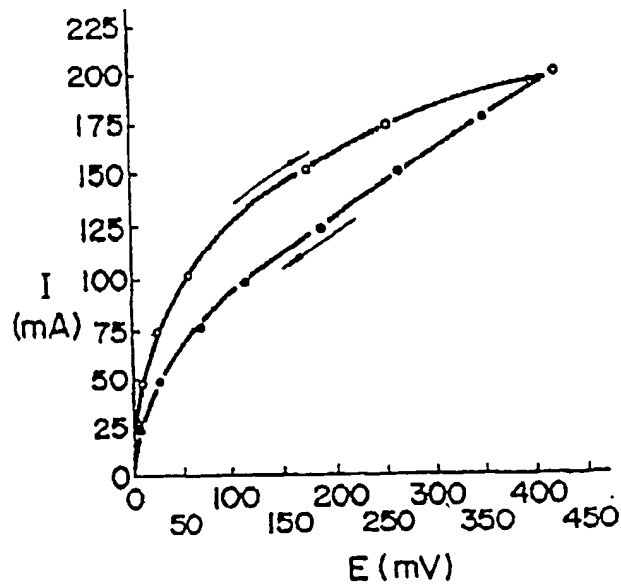
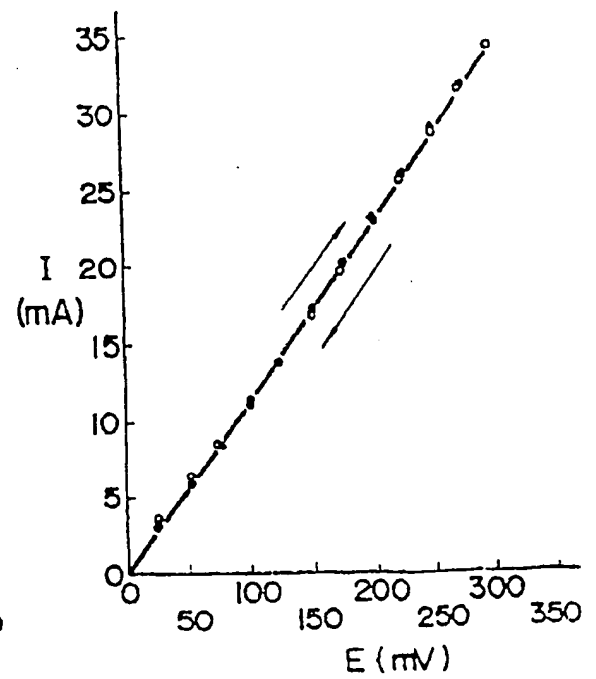
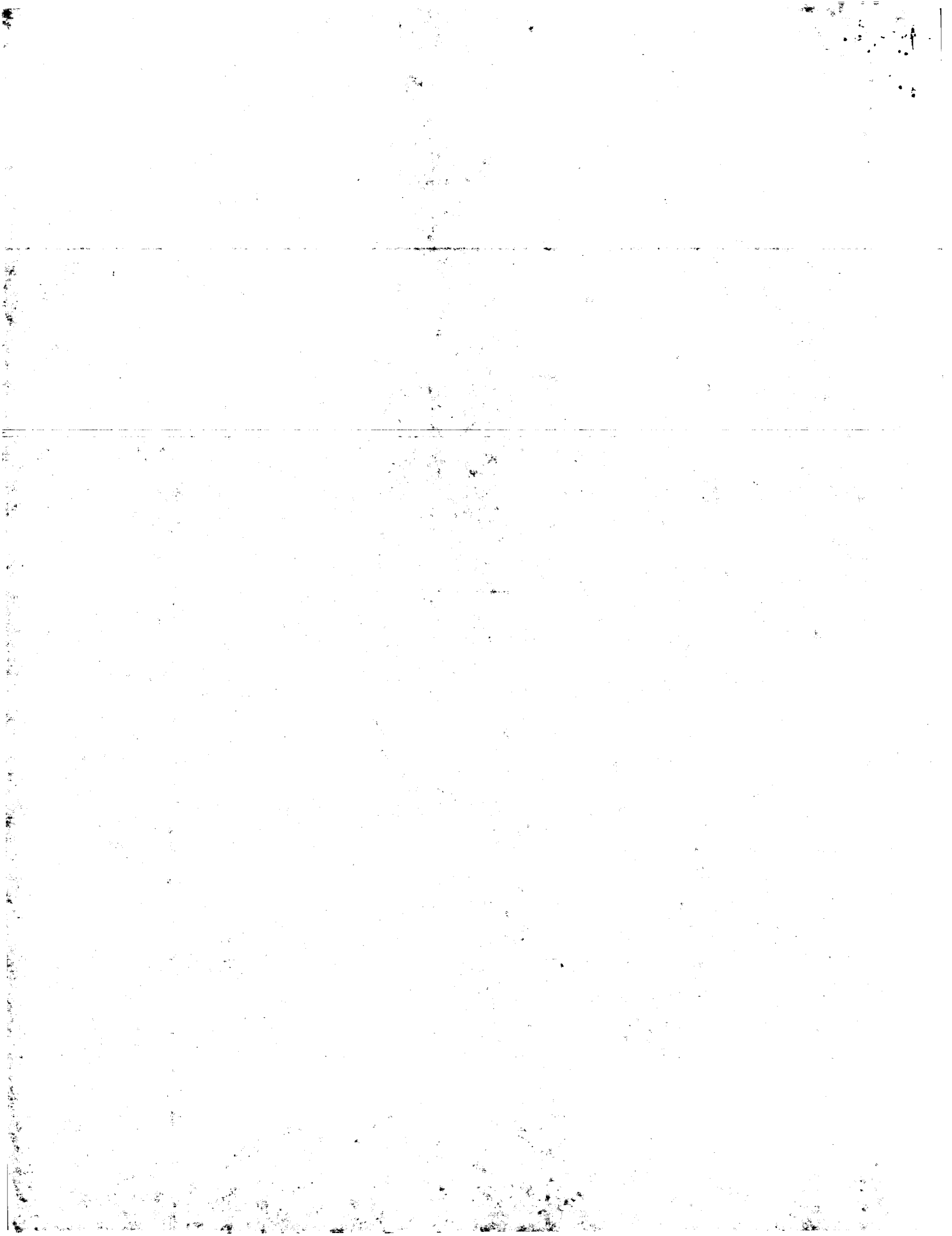


FIG. 6





**EUROPEAN PATENT APPLICATION**

Application number: 88310943.1

Int. Cl.<sup>5</sup> G01N 11/16

Date of filing: 18.11.88

Priority: 18.11.87 JP 289521/87

Date of publication of application:  
24.05.89 Bulletin 89/21

Designated Contracting States:  
CH DE FR GB IT LI SE

Date of deferred publication of the search report:  
28.03.90 Bulletin 90/13

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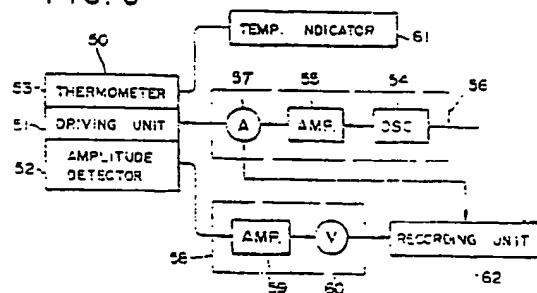
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**Vibration-type rheometer apparatus.**

A vibration-type rheometer including a vibration-type viscometer having a pair of vibrator subassemblies (4) which resonate as in a tuning fork. The pair of vibrator subassemblies (4) constituting a tuning fork vibrator each has at its free end a sensor plate (9) formed from a thin metal plate placed into a sample (35) to be measured and are driven at the same frequency with phases opposite by electromagnetic driving means (12, 13) together with the sensor plate (9). A control unit (56) is provided for supplying a driving current having a magnitude which changes steplessly and continuously to the electromagnetic driving means (12, 13) in order to change with time the vibration-inducing force in the pair of vibrator subassemblies (4). The amplitude of vibration of the vibrator subassemblies (4) which changes with the viscous resistance received by the sensor plates from the sample is electrically detected, and the detected value is sent to the record-

ing means (62) together with the value of the driving current to thereby indicate the behaviour of the sample (35) with respect to the external force which changes by time.

**FIG. 3**





European Patent  
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# EUROPEAN SEARCH REPORT

Application Number

EP 88 31 0943

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
D,A	EP-A-0 233 408 (CHICHIBU CEMENT K.K.) * Claims 1,2; Figure 1 * ---	1	G 01 N 11/16
A	INDUSTRIAL LABORATORY, vol. 50, no. 7, July 1984, pages 669-671, Plenum Publishing Corp., New York, US; V.N. GLADKII et al.: "Vibration viscosimeter" * Page 670, paragraph 2 - page 671, paragraph 1; figure 2 * ---	1	
A	CONTROL AND INSTRUMENTATION, vol. 14, no. 10, October 1982, pages 47,49, London, GB: "A fresh slant on the rheogoniometer" * Page 48, left-hand column * ---	1,3	
A	US-A-3 722 262 (GILINSON) * Columns 6,7, claims 1-3; figure 2A * -----	3-6	
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			G 01 N 11/00
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 27-11-1989	Examiner MILLS J.
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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